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IS 15694 (2006): Potassium iodate, food grade [FAD 8: Food Additives]



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भारतीय मानक

पोटेशियम आयोडेट, खाद्य ग्रेड — विशिष्टि

Indian Standard

POTASSIUM IODATE, FOOD GRADE —
SPECIFICATION

ICS 67.220.20

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

Potassium iodate is widely used for iodization of edible common salt. This standard has been prepared to cover the purity and identification of potassium iodate. This is extremely important, for it is extremely difficult to detect the impurity once it has been added to processed foods.

The requirement of foodgrade potassium iodate as given in this standard was earlier a part of IS 13057 : 1991 'Specification for potassium iodate', where in 3 grades — analytical, food and technical were specified. Through an amendment to IS 13057 in 2003, the foodgrade has been deleted from IS 13057 and, therefore, a separate standard has been formulated to cover the foodgrade potassium iodate.

In the preparation of this standard, due consideration has been given to the provisions of the *Prevention of Food Adulteration Act*, 1954 and Rules framed thereunder. However, this standard is subject to restrictions imposed under this Act, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

POTASSIUM IODATE, FOOD GRADE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for potassium iodate, food grade.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
1070 : 1992	Reagent grade water (<i>third revision</i>)
1699 : 1995	Methods of sampling and test for food colours (<i>second revision</i>)
4905 : 1968	Methods for random sampling
5741 : 1970	Method for determination of pH

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white crystalline powder, free from impurities.

3.2 The material shall also conform to the requirements given in Table 1.

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be packed in well-filled containers with minimum access to moisture. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage

The material shall be stored in a cool, dry place so as to avoid excessive exposure to heat.

4.3 Marking

4.3.1 Each container shall be legibly and indelibly marked with the following information:

- Name of the product including the words 'Food Grade';
- Name and address of the manufacturer;
- Source of manufacture;
- Minimum net contents;
- Date of manufacture;
- Batch or code number;
- The words 'Best before
- Any other requirements as specified under the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977* and the *Prevention of Food Adulteration Act, 1954* and Rules framed thereunder.

Table 1 Requirements for Potassium Iodate

(Clause 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Clause No. IS 1699
(1)	(2)	(3)	(4)	(5)
i)	Potassium iodate (as KIO_3), percent by mass, <i>Min</i>	99.8	A-2	—
ii)	Matter insoluble in water, percent by mass, <i>Max</i>	0.10	A-3	—
iii)	Bromate, bromide, chlorate and chloride (as Cl), percent by mass, <i>Max</i>	0.01	A-4	—
iv)	Iodide (as I), percent by mass, <i>Max</i>	0.002	A-5	—
v)	Sulphate (as SO_4), percent by mass, <i>Max</i>	0.02	A-6	—
vi)	Loss on drying, percent by mass, <i>Max</i>	0.1	A-8	—
vii)	Iron, mg/kg, <i>Max</i>	3	A-7	—
viii)	Heavy metals (as Pb), mg/kg, <i>Max</i>	10	—	16
ix)	Arsenic, mg/kg, <i>Max</i>	3	—	15
x)	pH (5 percent solution)	Neutral	A-9	—

4.3.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

4.3.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Representative samples of the material shall be drawn and the criteria for conformity to this standard shall be established according to the method prescribed in Annex B.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(Clause 3.2, Table 1)

METHOD OF TEST FOR POTASSIUM IODATE

A-1 PREPARATION OF SAMPLE

A-1.1 Sample for determination of potassium iodate content shall be the residual material obtained at the end of the test for loss on drying.

A-1.2 Sample for Other Tests

Crush 100 g of the sample so that it passes through 1.00 mm IS Sieve, and keep it in a clean glass-stoppered weighing bottle in a desiccator for subsequent tests.

A-2 DETERMINATION OF POTASSIUM IODATE

A-2.1 Reagents

A-2.1.1 *Potassium Iodide*

A-2.1.2 *Hydrochloric Acid* — Approximately 5 N.

A-2.1.3 *Standard Sodium Thiosulphate Solution* — 0.1 N.

A-2.1.4 *Starch Indicator Solution* — Mix 0.5 g of soluble starch with 5 ml of cold water. Add this mixture, with stirring, to 95 ml of boiling water and heat in a boiling water bath for 5 min.

A-2.2 Procedure

Dissolve 1.5 g of the dried material (at 130°C) in water and dilute to 500 ml. To 50 ml of this solution add 3 g of potassium iodide and 10 ml of dilute hydrochloric acid and titrate the liberated iodine with 0.1 N standard sodium thiosulphate solution, using starch as indicator. Perform a blank determination and make necessary correction, if any.

A-2.3 Calculation

Potassium iodate (as KIO_3),
percent by mass = $3.567 \frac{(V_1 - V_2)}{M}$

where

V_1 = volume in ml of standard sodium thiosulphate solution used in titrating the sample,

V_2 = volume in ml of standard sodium thiosulphate solution used in titrating the blank, and

M = mass in g of the sample taken for the test.

A-3 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure

Dissolve 20 g of the sample (*see* A-1.2) in about 400 ml of warm water. Cool the solution and set it aside for 1 h. Filter through a weighed sintered glass crucible (G No. 4). Wash the residue with water and dry to constant mass at 105°C.

A-3.2 Calculation

Matter insoluble in water,
percent by mass = $\frac{M_1 \times 100}{M_2}$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the sample taken for the test.

A-4 TEST FOR BROMATE, BROMIDE, CHLORATE AND CHLORIDE

A-4.1 Reagents

A-4.1.1 Sulphur Dioxide

A-4.1.2 Dilute Ammonia Solution — Approximately 5 N. Add 280 ml of strong ammonia solution (AR or GR grade, rd 0.88) to sufficient water to make up the volume to 1 000 ml.

A-4.1.3 Silver Nitrate Solution — 0.1 N. Dissolve 16.087 g of silver nitrate (AR or GR grade) in sufficient water to make up the volume to 100 ml.

A-4.1.4 Dilute Nitric Acid — Approximately 5 N. Add 320 ml of nitric acid (AR or GR grade, rd 1.42) to sufficient water and make up the volume to 1 000 ml.

A-4.1.5 Standard Chloride Solution — Dissolve 0.164 g of sodium chloride (NaCl) in water and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of chloride (as Cl).

A-4.2 Procedure

A-4.2.1 Dissolve 0.5 g of the sample in 50 ml of water and pass sulphur dioxide into the solution until the precipitate of iodine redissolves. Evaporate the solution to about 2 ml, cool, add 6 ml of dilute ammonia solution, transfer to a stoppered test tube and dilute to 15 ml with water. Add 15 ml of silver nitrate solution, shake well and filter. To 15 ml of filtrate, add 30 ml of water and 5 ml of dilute nitric acid. Any opalescence produced shall not be greater than the standard opalescence produced as in A-4.2.2.

A-4.2.2 Standard Opalescence

To 50 ml of water, add 0.5 ml of standard chloride solution, 1 ml of dilute nitric acid and 1 ml of silver nitrate solution, mixing after each addition. Use as comparison standard after 5 min.

A-5 TEST FOR IODIDE

A-5.1 Reagents

A-5.1.1 Dilute Sulphuric Acid

A-5.1.2 Starch Indicator Solution — Triturate 1 g of arrowroot starch with 10 ml of cold water and pour slowly, with constant stirring, into 200 ml of boiling water. Boil the mixture until a thin translucent fluid is obtained. Allow to settle and use only the clear, supernatant liquid. Prepare this solution fresh.

A-5.2 Procedure

Dissolve 1 g of the material in 10 ml of water and add 1 ml of dilute sulphuric acid and 1 drop of starch solution. No blue colour shall be formed.

A-6 TEST FOR SULPHATE

A-6.1 Method A (Turbidimetric Method)

A-6.1.1 Reagents

A-6.1.1.1 Industrial methylated spirit (absolute, 74 OP)

A-6.1.1.2 Industrial methylated spirit (20 percent) — Dilute 20 ml of absolute industrial methylated spirit with water to produce a final volume of 100 ml.

A-6.1.1.3 Barium chloride solution — Approximately 1 N. Dilute 430 ml of hydrochloric acid (AR or GR grade, rd 1.18) with sufficient water to make up the volume to 1 000 ml.

A-6.1.1.4 Standard sulphate solution — Dilute 20.8 ml of 0.1 N sulphuric acid with sufficient water to make up the volume to 1 000 ml. This solution contains 0.1 mg of SO_4 per ml.

A-6.1.2 Procedure

A-6.1.2.1 Dissolve 1 g of the sample in 40 ml of water, add 0.6 g of hydroxy ammonium chloride and boil till the liberated iodine is expelled. Cool, dilute to 40 ml with water, add 10 ml of dilute hydrochloric acid. Mix the resulting solution after adding 1 ml of barium chloride solution, and allow to stand for 1 h. Any turbidity produced shall not be greater than the standard turbidity produced as in A-6.1.2.2.

A-6.1.2.2 Standard turbidity

Take 2 ml of standard sulphate solution. Add 37 ml of water, 10 ml of absolute industrial methylated spirit, 1 ml of dilute hydrochloric acid and mix. Add 1 ml of barium chloride solution, mix immediately and allow to stand for 1 h.

A-6.2 Method B (Gravimetric Method)

A-6.2.1 Reagents

A-6.2.1.1 Dilute hydrochloric acid — Approximately 4 N.

A-6.2.1.2 Barium chloride — Approximately 10 percent.

A-6.2.2 Procedure

Dissolve 1 g of the sample in 40 ml of water, add 0.6 g of hydroxy ammonium chloride and boil till the liberated iodine is expelled. Cool and dilute to 400 ml with water. Add one drop of methyl orange and about 10 ml of dilute hydrochloric acid till it becomes pink and then boil. To the boiling solution add barium chloride solution drop by drop so that the addition is in slight excess and continue boiling for 4 min to

obtain a granular precipitate. Allow it to stand for 4 h and filter through a weighed sintered glass crucible (G No. 4). Wash the precipitate till it is free from chloride and dry to constant mass at 105 to 110°C. Alternatively, filter through Whatman filter paper No. 42 and wash till it is free from chloride. Ignite and weigh as barium sulphate.

A-6.2.3 Calculation

$$\text{Sulphate (as SO}_4\text{), percent by mass} = \frac{41.13 M_1}{M_2}$$

where

M_1 = mass in g of the barium sulphate precipitate, and

M_2 = mass in g of the sample taken for the test.

A-7 TEST FOR IRON

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinder* — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 *Dilute Sulphuric Acid* — Approximately 5 N. Pour 136 ml of sulphuric acid (AR grade, 36 N) in sufficient quantity of water, mix and allow to cool. Make the volume to 1 000 ml.

A-7.2.2 *Dilute Hydrochloric Acid* — Approximately 5 N. Dilute 430 ml of hydrochloric acid (AR grade, rd 1.18) with sufficient water to make up the volume to 1 000 ml.

A-7.2.3 *Potassium Permanganate Solution* — 0.1 N.

A-7.2.4 *Ammonium Thiocyanate Solution* — Approximately 7.5 N. Dissolve 570 g of ammonium thiocyanate (AR grade) in sufficient water and make up the volume to 1 000 ml.

A-7.2.5 *Amyl Acetate* — AR or GR grade.

A-7.2.6 *Amyl Alcohol* — AR or GR grade.

A-7.2.7 *Standard Iron Solution* — Dissolve 0.070 2 g of ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$] in water, and add 10 ml of dilute sulphuric acid (10 percent v/v). Dilute to exactly 100 ml in a volumetric flask. Take 25 ml of this solution into a 250 ml volumetric flask and dilute to the mark with water. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-7.3 Procedure

Dissolve 3 g of the sample in a mixture of 30 ml of water and 5 ml of dilute sulphuric acid and pass sulphur

dioxide through the solution until the precipitate of iodine redissolves. Evaporate the solution to dryness on a hot plate, mix the residue with 10 ml of dilute hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue by warming with a mixture of 3 ml of dilute hydrochloric acid and 27 ml of water. Retain 20 ml for the heavy metals test and to 10 ml apply the iron test as given in A-7.3.1.

A-7.3.1 Take 10 ml of solution in a 50 ml Nessler cylinder obtained in A-7.3, add 1 drop of potassium permanganate solution and mix. Add 5 ml of ammonium thiocyanate solution and 10 ml of a mixture of equal volumes of amyl alcohol and amyl acetate. Shake vigorously and allow to separate. Any red colour produced in the upper layer shall not be deeper than that produced in the upper layer of the standard as in A-7.3.2.

A-7.3.2 To 1 ml of standard iron solution in a 50 ml Nessler cylinder, add 1 ml of dilute hydrochloric acid and dilute with water to the same volume as the acidified solution of the test sample. Add 1 drop of potassium permanganate solution and mix. Add 5 ml of a mixture of equal volumes of amyl alcohol and amyl acetate. Shake vigorously and allow to separate.

A-8 DETERMINATION OF LOSS OF MASS ON DRYING AT 130°C

A-8.1 Apparatus

A-8.1.1 *Weighing Bottle* — Approximately 100 ml capacity, 75 mm in diameter, with ground glass stopper.

A-8.1.2 *Electric Oven* — Capable of maintaining a temperature of $130 \pm 2^\circ\text{C}$.

A-8.2 Procedure

Weigh 5 g of the material to the nearest 0.1 mg. Spread thin layers of the material in the weighing bottle which shall be previously dried for 30 min in the oven controlled at a temperature of 130°C , cooled in a desiccator and weighed. Place the weighing bottle in the oven with its ground glass stopper placed slantwise. Gradually heat the oven from the starting temperature (which should not exceed 100°C) to $130 \pm 2^\circ\text{C}$ and maintain this temperature for 1 h. Remove the weighing bottle, place it in a desiccator and cool. After cooling to room temperature, weigh it again with the stopper in position.

A-8.3 Calculation

$$\text{Loss of mass on drying at } 130^\circ\text{C, percent by mass} = \frac{(M_1 - M_2) \times 100}{M}$$

where

M_1 = mass in g of the material and weighing bottle before heating,

M_2 = mass in g of the material and weighing bottle after heating, and

M = mass in g of the material taken for the test.

A-9 DETERMINATION OF pH OF AQUEOUS SOLUTION

Dissolve 5.0 g of the material in 100 ml of freshly boiled and cooled water (the water shall be protected with soda lime tube while cooling). Determine the pH of the solution using a suitable pH meter (*see* IS 5741).

ANNEX B

(Clause 5)

SAMPLING OF POTASSIUM IODATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.2 The samples shall be placed in suitable clean and dry containers.

B-1.3 The sample containers shall be of such size that they are almost completely filled by sample.

B-1.4 Each sample containers shall be sealed airtight after filling and marked with full details of sampling and the date of sampling.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.2 Samples shall be tested from each lot separately for ascertaining conformity of the material to the requirements of this specification. The number of containers to be selected from a lot (n) shall depend on the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling
(Clause B-2.2)

Lot Size N	Number of Containers to be Selected n
(1)	(2)
4-50	3
51-100	4
101-150	5
151-300	7
301 and above	10

NOTE — When the size of the lot is 3 or less, the number of containers to be selected and the criterion for judging the conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 These containers shall be selected at random from the lot. For random selection procedure, guidelines can be had from IS 4905. In case IS 4905 is not available, the following procedure shall be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them 1, 2, 3, ..., etc, up to r and so on, where r is the integral part of N/n . Every r th container thus counted shall be taken out.

B-3 PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

B-3.1 For drawing samples, an auger shall be used. Introduce the auger in different parts of each container to draw an appropriate quantity of the sample from each container.

B-3.2 Out of portions collected from all the selected containers, an equal quantity of the material shall be

taken out and mixed together to form a composite sample of about 250 g. The composite sample so formed shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the sample from each container shall be divided into three equal parts and each such separate part shall constitute an individual sample. One set of individual samples (representing the n containers selected) shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.4 All the individual and the composite samples shall be immediately transferred to separate glass bottles which shall be labelled with full identification particulars of the samples.

B-3.5 The referee samples shall bear the seal of both the purchaser and the supplier, to be used in case of dispute between the two.

B-4 NUMBER OF TESTS

Tests for potassium iodate content, matter insoluble in water and loss on drying shall be conducted on each of the individual samples and tests for all other

requirements given in 3.1 and Table 1 shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

A lot shall be considered as conforming to the requirements of this specification if the stipulations laid down in **B-5.1** and **B-5.2** are fulfilled.

B-5.1 For Potassium Iodate

From the individual test results for potassium iodate content the mean (X) and the range (R) or mean range (Rm) shall be calculated. (Range is the difference between the maximum and the minimum of the test results). When the sample size is 10, the test results shall be arranged in two groups of 5 each in the order of their occurrence and the range for each group computed. R will be the mean of these two R 's. The lot shall be deemed satisfactory for this characteristic if $(X - 0.6 R)$ or $(X - 0.6 Rm)$ is greater than or equal to the minimum requirement specified in Table 1.

B-5.2 For Other Characteristics

The test results for all other characteristics prescribed in 3.1 and Table 1 shall satisfy the relevant requirements.

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